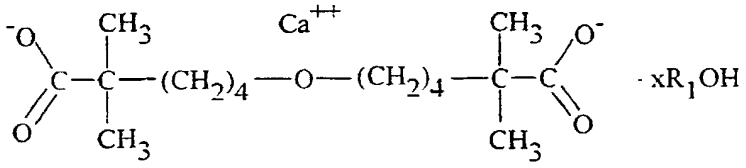
CLAIMS

What is claimed is:

1. A compound that is 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt of Formula II:



II

wherein R_1 is H or lower alkyl and x is a number from 0 to 10.

2. The compound 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt.

3. The compound 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt hydrate.

4. The compound 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt Crystal Form 1 having an x-ray powder diffraction pattern substantially comprising:

#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	6.760	13.0648	5106	100.0	1497	100.0	0.234
2	8.183	10.7953	1743	34.1	435	29.1	0.200
3	8.560	10.3207	1866	36.5	543	36.3	0.233
4	9.239	9.5638	234	4.6	29	1.9	0.096
5	9.760	9.0546	972	19.0	220	14.7	0.181
6	10.569	8.3634	156	3.1	12	0.8	0.061
7	11.141	7.9353	178	3.5	29	1.9	0.130
8	13.760	6.4304	266	5.2	46	3.1	0.138
9	15.599	5.6761	338	6.6	63	4.2	0.148
10	16.740	5.2917	433	8.5	64	4.3	0.118
11	17.420	5.0866	1890	37.0	689	46.0	0.291
12	20.639	4.3000	523	10.2	128	8.5	0.196
13	21.391	4.1505	188	3.7	20	1.3	0.085
14	22.139	4.0119	445	8.7	74	4.9	0.132
15	31.559	2.8326	270	5.3	24	1.6	0.070

5. The compound 6-(5-carboxy-5-methyl-hexyloxy)-2.2-dimethylhexanoic acid monocalcium salt having a ^{13}C NMR (solid state) in ppm of: 189.6; 186.2; 71.4; 43.4; 30.1; 28.4; 25.2; 23.1.

6. The compound 6-(5-carboxy-5-methyl-hexyloxy)-2.2-dimethylhexanoic acid monocalcium salt having a ^{13}C NMR peak at 25.2 ppm.

7. The crystalline compound of Claim 1. wherein said compound comprises 6-(5-carboxy-5-methyl-hexyloxy)-2.2-dimethylhexanoic acid mono-calcium salt ethanol solvate. wherein R_1 is ethyl.

8. The crystalline compound 6-(5-carboxy-5-methyl-hexyloxy)-2.2-dimethylhexanoic acid monocalcium salt ethanol solvate having an x-ray powder diffraction pattern substantially comprising:

#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	6.899	12.8028	13186	100.0	3025	100.0	0.184
2	8.261	10.6945	5221	39.6	931	30.8	0.143
3	8.838	9.9969	2057	15.6	482	15.9	0.187
4	11.061	7.9927	785	6.0	160	5.3	0.163
5	12.100	7.3086	1355	10.3	150	4.9	0.088
6	13.619	6.4964	450	3.4	89	2.9	0.157
7	17.677	5.0132	753	5.7	126	4.2	0.134
8	18.180	4.8755	2011	15.3	588	19.4	0.234
9	20.840	4.2588	439	3.3	40	1.3	0.072
10	21.334	4.1615	427	3.2	67	2.2	0.125

9. The crystalline compound of Claim 8 having a ^{13}C NMR (solid state) in ppm of: 189.9; 186.7; 71.6; 58.5; 43.2; 29.9; 23.5.

10. The compound 6-(5-carboxy-5-methyl-hexyloxy)-2.2-dimethylhexanoic acid monocalcium salt. ethyl alcohol solvate. having a ^{13}C NMR peak at 58.5 ppm.

11. The crystalline compound 6-(5-carboxy-5-methyl-hexyloxy)-2.2-dimethylhexanoic acid mono-calcium salt methanol solvate.

12. The crystalline compound of Claim 11. having an x-ray powder diffraction pattern substantially comprising:

#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	6.896	12.8072	11991	100.0	2593	100.0	0.173
2	8.339	10.5940	2046	17.1	334	12.9	0.131
3	9.219	9.5853	1438	12.0	281	10.8	0.156
4	10.280	8.5979	632	5.3	180	6.9	0.227
5	11.320	7.8105	1079	9.0	322	12.4	0.238
6	15.800	5.6044	463	3.9	59	2.3	0.102
7	16.741	5.2913	432	3.6	38	1.4	0.069
8	18.160	4.8809	1260	10.5	599	23.1	0.380
9	18.702	4.7408	700	5.8	184	7.1	0.210
10	19.816	4.4766	589	4.9	94	3.6	0.127
11	21.724	4.0876	510	4.3	96	3.7	0.150

13. The crystalline compound of Claim 12 having a ^{13}C NMR (solid state) in ppm of: 189.6: 186.2: 71.4: 43.2: 29.6: 23.5.

14. The compound which is 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt 1-propyl alcohol solvate.

15. The crystalline compound of Claim 14 having an x-ray powder diffraction pattern substantially comprising:

#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	6.899	12.8025	12371	100.0	3495	100.0	0.226
2	7.843	11.2637	4815	38.9	1119	32.0	0.186
3	8.661	10.2009	1709	13.8	357	10.2	0.167
4	11.359	7.7833	771	6.2	141	4.0	0.146
5	12.300	7.1900	752	6.1	127	3.6	0.135
6	13.100	6.7528	517	4.2	37	1.0	0.057
7	18.262	4.8540	1945	15.7	596	17.1	0.245
8	20.721	4.2832	828	6.7	279	8.0	0.269
9	21.740	4.0847	573	4.6	146	4.2	0.203

16. The crystalline compound of Claim 15 having a ^{13}C NMR (solid state) in ppm of: 189.6; 186.2; 71.4; 43.2; 29.6; 23.5.

17. The compound which is 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid mono-calcium salt 2-propyl alcohol solvate.

18. The crystalline compound of Claim 17 having an x-ray powder diffraction pattern substantially comprising:

#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	6.918	12.7674	10028	100.0	2562	100.0	0.204
2	8.000	11.0427	3984	39.7	800	31.2	0.161
3	8.619	10.2506	1619	16.1	346	13.5	0.171
4	11.338	7.7981	658	6.6	68	2.6	0.082
5	11.718	7.5459	236	2.4	28	1.1	0.093
6	12.241	7.2243	761	7.6	131	5.1	0.138
7	15.382	5.7557	610	6.1	107	4.2	0.140
8	18.162	4.8803	1937	19.3	441	17.2	0.182
9	20.779	4.2713	853	8.5	222	8.6	0.208

19. The crystalline compound of Claim 14 having a ^{13}C NMR (solid state) in ppm of: 189.4; 187.7; 70.9; 69.4; 66.5; 63.8; 43.2; 35.0; 30.1; 23.8; 18.7; 14.3.

20. The compound 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt 2-propyl alcohol solvate having a ^{13}C NMR peak at 63.8, 18.7, or 14.3 ppm.

21. The compound which is 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt 1-butyl alcohol solvate.

22. The crystalline compound of Claim 21 having an x-ray powder diffraction pattern substantially comprising:

#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	7.060	12.5101	19609	100.0	4796	100.0	0.196
2	9.078	9.7332	3027	15.4	567	11.8	0.150
3	11.100	7.9644	924	4.7	164	3.4	0.142
4	16.361	5.4135	554	2.8	76	1.6	0.109
5	18.040	4.9133	2276	11.6	456	9.5	0.160
6	18.820	4.7112	1303	6.6	385	8.0	0.236
7	19.922	4.4532	1886	9.6	457	9.5	0.193
8	21.560	4.1183	853	4.4	205	4.3	0.191
9	22.281	3.9867	343	1.7	37	0.8	0.086
10	23.521	3.7793	450	2.3	107	2.2	0.189

23. The compound of Claim 21 having a ^{13}C NMR (solid state) in ppm of: 189.9; 186.0; 71.6; 43.2; 29.9; 23.8.

24. The compound 6-(5-carboxy-5-mentyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt Crystal Form 2 having an x-ray powder diffraction pattern substantially comprising:

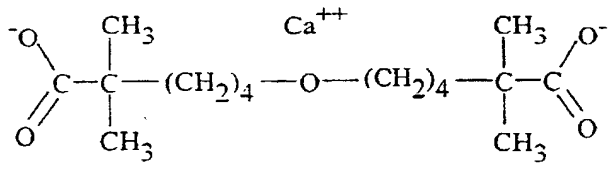
#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	7.259	12.1686	9283	100.0	2482	100.0	0.214
2	8.739	10.1100	4191	45.1	603	24.3	0.115
3	9.386	8.9628	967	10.4	161	6.5	0.133
4	11.659	7.5838	430	4.6	49	1.9	0.089
5	13.955	6.3408	305	3.3	58	2.3	0.151
6	14.220	6.2233	326	3.5	73	2.9	0.178
7	15.387	5.7537	278	3.0	19	0.7	0.053
8	16.461	5.3806	986	10.6	187	7.5	0.152
9	17.361	5.1039	1490	16.1	348	14.0	0.187
10	18.063	4.9069	1284	13.8	323	13.0	0.201
11	19.302	4.5947	871	9.4	166	6.7	0.152
12	19.862	4.4664	686	7.4	142	5.7	0.166
13	20.200	4.3923	457	4.9	103	4.1	0.179
14	21.178	4.1918	656	7.1	97	3.9	0.117
15	21.641	4.1031	167	1.8	6	0.2	0.029
16	22.300	3.9833	794	8.6	192	7.7	0.193
17	23.218	3.8278	247	2.7	23	0.9	0.071
18	24.100	3.6897	183	2.0	34	1.3	0.145
19	25.481	3.4928	487	5.2	141	5.7	0.231
20	28.800	3.0974	134	1.4	14	0.6	0.083
21	29.297	3.0459	259	2.8	28	1.1	0.084
22	30.700	2.9099	287	3.1	20	0.8	0.055

25. The crystalline compound of Claim 24 having a ^{13}C NMR (solid state) in ppm of 190.9; 189.6; 186.2; 120.4; 72.7; 44.7; 44.2; 43.0; 42.3; 39.3; 37.9; 31.8; 30.9; 29.6; 27.7; 26.2; 25.3; 24.0; 22.9; 21.5; and 20.2.

26. The compound 6-(5-carboxy-5-mentyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt Crystal Form 2 having a ^{13}C NMR peak at 72.7, 44.7, or 26.2 ppm.

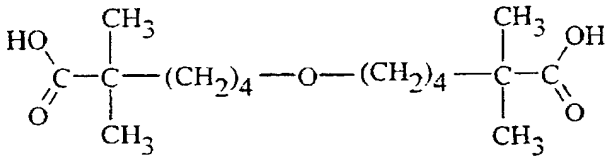
27. The compound of Claim 1. wherein said crystalline structure contains from approximately 0.1 to approximately 1.0 water molecules per salt ion.

28. A method for preparing a stable crystalline compound of Formula II:



II

wherein R_1 is lower alkyl and x is a number from 0 to 10. comprising reacting a compound of Formula I



I

with calcium oxide in an alkanol organic solvent of the formula R_1OH to yield a solid product: and drying the solid product to obtain the monocalcium dicarboxylate ether salt of the compound of Formula II having a stoichiometric ratio of calcium to dicarboxylate of approximately 1:1.

29. The method of Claim 28. wherein the organic solvent is a C_1-C_{12} alcohol.

30. The method of Claim 28. wherein the C_1-C_{12} alcohol is essentially anhydrous.

31. The method of Claim 28. wherein the alcohol is a C_1-C_4 alkanol.

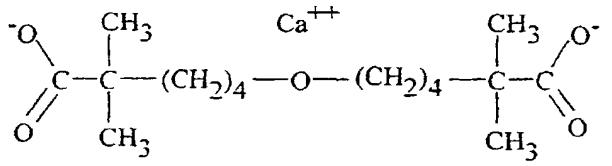
32. The method of Claim 28 further comprising the step of introducing a work-up solvent into the organic alcohol solvent. wherein the work-up solvent causes at least a portion of the monocalcium dicarboxylate ether salt to precipitate from the organic alcohol solvent.

33. The method of Claim 32 wherein the work-up solvent is methyl *tert*-butyl ether.

34. The method of Claim 32 further comprising the step of filtering the solid product from the organic solvent prior to drying.

35. The method of Claim 32 further comprising the step of washing the solid product with the organic work-up solvent subsequent to filtering.

36. A method for preparing a crystalline hydrate of the Formula II



II

wherein R_1 is H and x is a number from 0 to 10. comprising reacting an alcohol solvate of Formula II where R_1 is lower alkyl with water.

37. The method of Claim 36 wherein the solid product contains between approximately 0.1 and approximately 1.0 equivalents of water per equivalent of the monocalcium dicarboxylate ether salt subsequent to said filtering step and said drying step.

38. The method of Claim 28 wherein said reacting step occurs at a temperature between about 15°C and the reflux point of the alkanol organic solvent at standard pressure.

39. The method of Claim 28 wherein said reacting step occurs at a temperature between the reflux point of the alkanol organic solvent and about 150°C at a pressure above standard pressure.

40. A method of converting the compound 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt hydrate Crystal Form 1 into the compound 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic

acid monocalcium salt hydrate Crystal Form 2, said method comprising the steps of: exposing the Crystal Form 1 to water; agitating the Crystal Form 1 and water; heating the Crystal Form 1 and water for sufficient time for a conversion to occur so as to yield Crystal Form 2; and drying the solid product to obtain the 6-(5-carboxy-5-methylhexyloxy)-2,2-dimethylhexanoic acid monocalcium salt hydrate Crystal Form 2, wherein the Crystal Form 2 has a stoichiometric ratio of calcium to dicarboxylate form of the compound of 1:1.

41. The method of Claim 40 further comprising the step of filtering the Crystal Form 2 from the water prior to said drying step.

42. A pharmaceutical composition comprising the compound 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt together with one or more pharmaceutically acceptable diluents, carriers or excipients.

43. A pharmaceutical composition comprising a crystalline form 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt together with one or more pharmaceutically acceptable diluents, carriers or excipients.

44. A pharmaceutical composition comprising 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt hydrate Crystal Form 1 together with one or more pharmaceutically acceptable diluents, carriers or excipients.

45. A pharmaceutical composition comprising 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt hydrate Crystal Form 2 together with one or more pharmaceutically acceptable diluents, carriers or excipients.

46. The use of a compound as set forth in Claim 1 for the treatment of vascular disease.

47. The use of a compound as set forth in Claim 1 for the treatment of diabetes.

48. A compound according to Claim 1 substantially as described herein in any of the examples.

49. A method of treating a vascular disease in a patient in need thereof, said method comprising administering to the patient a therapeutically effective amount of 6-(5-carboxy-5-methyl-hexyloxy)-2,2-dimethylhexanoic acid monocalcium salt.

50. A method according to Claim 49, wherein the compound is Crystal Form I having an x-ray powder diffraction pattern substantially comprising:

#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	6.760	13.0648	5106	100.0	1497	100.0	0.234
2	8.183	10.7953	1743	34.1	435	29.1	0.200
3	8.560	10.3207	1866	36.5	543	36.3	0.233
4	9.239	9.5638	234	4.6	29	1.9	0.096
5	9.760	9.0546	972	19.0	220	14.7	0.181
6	10.569	8.3634	156	3.1	12	0.8	0.061
7	11.141	7.9353	178	3.5	29	1.9	0.130
8	13.760	6.4304	266	5.2	46	3.1	0.138
9	15.599	5.6761	338	6.6	63	4.2	0.148
10	16.740	5.2917	433	8.5	64	4.3	0.118
11	17.420	5.0866	1890	37.0	689	46.0	0.291
12	20.639	4.3000	523	10.2	128	8.5	0.196
13	21.391	4.1505	188	3.7	20	1.3	0.085
14	22.139	4.0119	445	8.7	74	4.9	0.132
15	31.559	2.8326	270	5.3	24	1.6	0.070

51. A method according to Claim 49, wherein the compound is Crystal Form 2 having an x-ray powder diffraction pattern substantially comprising:

#	2-Theta	d(A)	Peak	P%	Area	Area%	FWHM
1	7.259	12.1686	9283	100.0	2482	100.0	0.214
2	8.739	10.1100	4191	45.1	603	24.3	0.115
3	93860	8.9628	967	10.4	161	6.5	0.133
4	11.659	7.5838	430	4.6	49	1.9	0.089
5	13.955	6.3408	305	3.3	58	2.3	0.151
6	14.220	6.2233	326	3.5	73	2.9	0.178
7	15.387	5.7537	278	3.0	19	0.7	0.053
8	16.461	5.3806	986	10.6	187	7.5	0.152
9	17.361	5.1039	1490	16.1	348	14.0	0.187
10	18.063	4.9069	1284	13.8	323	13.0	0.201
11	19.302	4.5947	871	9.4	166	6.7	0.152
12	19.862	4.4664	686	7.4	142	5.7	0.166
13	20.200	4.3923	457	4.9	103	4.1	0.179
14	21.178	4.1918	656	7.1	97	3.9	0.117
15	21.641	4.1031	167	1.8	6	0.2	0.029
16	22.300	3.9833	794	8.6	192	7.7	0.193
17	23.218	3.8278	247	2.7	23	0.9	0.071
18	24.100	3.6897	183	2.0	34	1.3	0.145
19	25.481	3.4928	487	5.2	141	5.7	0.231
20	28.800	3.0974	134	1.4	14	0.6	0.083
21	29.297	3.0459	259	2.8	28	1.1	0.084
22	30.700	2.9099	287	3.1	20	0.8	0.055